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Kinetic and thermodynamic studies of U(VI) adsorption using Dowex-Marathon resin

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Abstract The kinetics and thermodynamics of U(VI) adsorption on Dowex-Marathon anionic resin have been studied. The process was well described by the pseudo-second order kinetic model for 0.02–0.1 M U(VI) concentration. The activation energy of U(VI) adsorption on Dowex-Marathon resin was determined: 6.62 ± 0.16 kJ/mol, indicating that the adsorption has a low potential barrier and corresponds to a physisorption. The thermodynamic parameter were evaluated: $\Delta G° = (-6.37)/(-9.23)$ kJ/mol, for temperature range: 20–60 °C; $\Delta H° = 14.57$ kJ/mol and $\Delta S° = 71.48$ J/mol K, the U(VI) adsorption being spontaneous and endothermic.

Keywords U(VI) · Adsorption · Dowex-Marathon resin · Kinetics · Isotherms · Thermodynamics

Introduction

Nuclear industry provides high quantity of wastewaters containing uranium, so that is important to find a method in order to reduce it. The sorption of U(VI) on adsorbents is one of the frequently method used for this purpose. Uranium (VI) can be adsorbed on organozeolites [1], volcanic rocks [2], Sorrel cements [3], ion exchange resins [4–7]. On the other hand, the sorption of U(VI) is important in isotope separation by isotope exchange chromatography. Shimokawa initiated the study on the uranium enrichment based on $U(IV)_{(R)}$–$U(VI)_{(S)}$ exchange reaction by using cationic exchange resin [8]. In 1978, Fujii reported the first successful enrichment by using anion exchange resin [9].

In uranium isotope separation by oxidation–reduction chromatography, for uranium band displacement in a separation column, filled with an anionic resin in Cl^- form, an oxidant $FeCl_3$ and a reductant $TiCl_3$ are used [10]. In the rear boundary of uranium band, uranyl ions adsorbed on anionit in chlorocomplexes form, are reduced to uranous ions with $TiCl_3$ from eluent; uranous ions flows through the resin bed, with the liquid phase, in the front of uranium band, uranous ions are oxidized to U(VI) by Fe(III), adsorbed on the resin in chlorocomplexes form. The isotope exchange reaction occurs repeatedly between U(VI) adsorbed on the resin and U(IV) from the solute, as the uranium band descends through the column. U(VI) forms negatively charged chlorocomplexes and is readsorbed, restoring the uranium band. The reactions that occur are [11]:

$$UO_{2(R)}^{2+} + 2Ti_{(S)}^{3+} \rightarrow U_{(S)}^{4+} + 2TiO_{(R)}^{2+} \quad \text{(in the rear boundary of uranium band)} \qquad (1)$$

$$U_{(S)}^{4+} + 2Fe_{(R)}^{3+} + 2H_2O \rightarrow UO_{2(R)}^{2+} + 2\,Fe_{(S)}^{2+} + 4H^+ \quad \text{(in the front of uranium band)} \qquad (2)$$

$$^{238}U(VI)_{(R)} + {}^{235}U(IV)_{(S)} \rightleftharpoons {}^{235}U(VI)_{(R)} + {}^{238}U(IV)_{(S)} \quad \text{(the isotope exchange reaction)} \qquad (3)$$

$UO_{2(R)}^{2+}$, $TiO_{(R)}^{2+}$, $Fe_{(R)}^{3+}$—chlorocomplexes of uranium, titanium and iron adsorbed on resin.

$Ti_{(S)}^{3+}$, $U_{(S)}^{4+}$, $Fe_{(R)}^{2+}$—aquo-chloro-complexes.

In order to find the conditions in which the rate of the reaction (1) is high enough, to lead to a net separation between $Ti(III)_{(S)}$ and $U(VI)_{(R)}$, which is needed in the ^{235}U separation column, we tried to determine the kinetics of this reaction. We found that the activation energy of

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$U(VI)_{(R)}$ reduction with $Ti(III)_{(S)}$, has a quite small value (12.39 ± 0.31 kJ/mol), indicating that we are in a diffusive domain, not in a kinetic one. In order to see if we really are in a diffusive domain, the study of $U(VI)_{(S)}$ adsorption on anionite Dowex-Marathon, was initiated.

Experimental

In the experiments, 0.02–0.1 M UO_2Cl_2 in 5 M HCl was used. Uranium solutions were prepared converting $UO_2(NO_3)_2 \cdot 6H_2O$ (Merck) in UO_2Cl_2 with concentrated HCl. Uranium (VI) concentration was determined after its reduction in Jones column to U(IV), by titration with $KMnO_4$ 10^{-2} N [12]; 1 ml of that solution is equivalent to 1.19034 mg uranium.

Hydrochloric solutions were prepared diluting 35–38 % HCl, pure P.A. (POCH SA, Poland) with appropriate volume of double distilled water.

The anionic resin used for adsorption experiments is Dowex-Maraton (Sigma–Aldrich, Germany), the characteristics of which are given in Table 1. This resin has a suitable value of capacity for U(VI) adsorption [13, 14].

The experiments were carried out by adding 10 g Dowex-Marathon resin (in Cl^- form by equilibration with 5 M HCl) and 100 ml UO_2Cl_2 in 5 M HCl with concentration ranging from 0.02 to 0.1 M, in a glass beaker. The mixture was stirred with a thermostated shaker (AREX Heating Magnetic Stirrer) at a constant speed of 200 rpm. Samples of 1 ml UO_2Cl_2 were taken from this solution in order to determine the time in which equilibrium was established. U(VI) was determinated after UO_2Cl_2 conversion into sulphate form, with concentrated H_2SO_4, followed by reduction in Jones column and titration with $KMnO_4$ 10^{-2} N. The amount of adsorbed uranium was estimated from the difference of uranium amounts in the solution before and after adsorption.

For all results presented in this paper, the experimental error is ±0.06 mg U.

Table 1 Dowex-Marathon resin characteristics

Name	Dowex-Marathon
Type	Macroporous, strong base
Appearance	Yellow-tan beads
Exchange capacity, in Cl^- form	1.1 meq/ml
Water retention capacity	62 %
Particle size	20–50 Mesh (0.8–0.32 mm)
Producer	Dow chemical Co, USA

Results and discussion

Influence of contact time and uranium initial concentration

In Fig. 1 the influence of contact time on U(VI) adsorption onto Dowex-Marathon resin is presented. The amount of uranium increased with time and U(VI) initial concentration and reached adsorption equilibrium within 40 min. For the further work, the adsorption equilibrium time has been taken as 40 min. For the obtained data it is clear that the kinetics of adsorption has two steps: an initial rapid step or external surface adsorption (the first 5 min) and a gradual adsorption step where intraparticle diffusion controls the adsorption rate until finally the equilibrium is reached. Similar results were reported for U(VI) adsorption onto Amberlite IRA-910 resins [7], cross-linked chitosan [15], beta type of akaganeite [16].

Influence of temperature

The amount of uranium adsorbed at equilibrium increases gradually with temperature. This behavior could be attributed to the acceleration of some originally slow adsorption steps, to formation of some new active sites on the surface of the adsorbent, or to transport against a concentration gradient and/or diffusion controlled transport across the energy barrier [17].

Adsorption isotherms

Langmuir isotherm (Fig. 2a) is derived from the assumption of monolayer coverage of adsorbate over a homogenous adsorbent surface [18]. The linear form of Langmuir isotherm equation is given as:

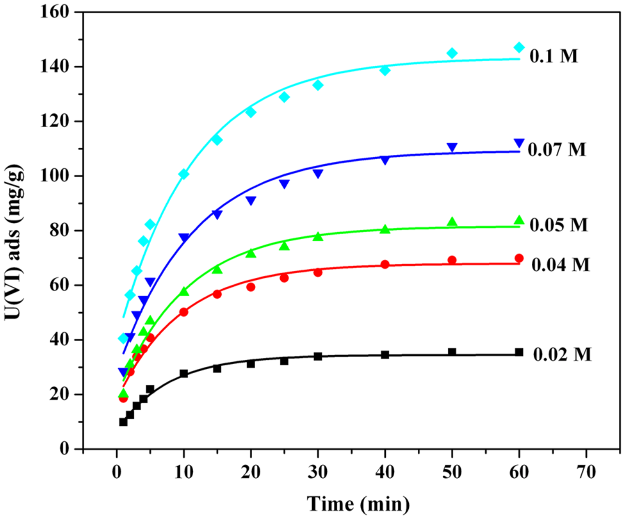

Fig. 1 Influence of contact time on U(VI) adsorption onto Dowex-Marathon resin, at 20 °C, for different U(VI) initial concentrations

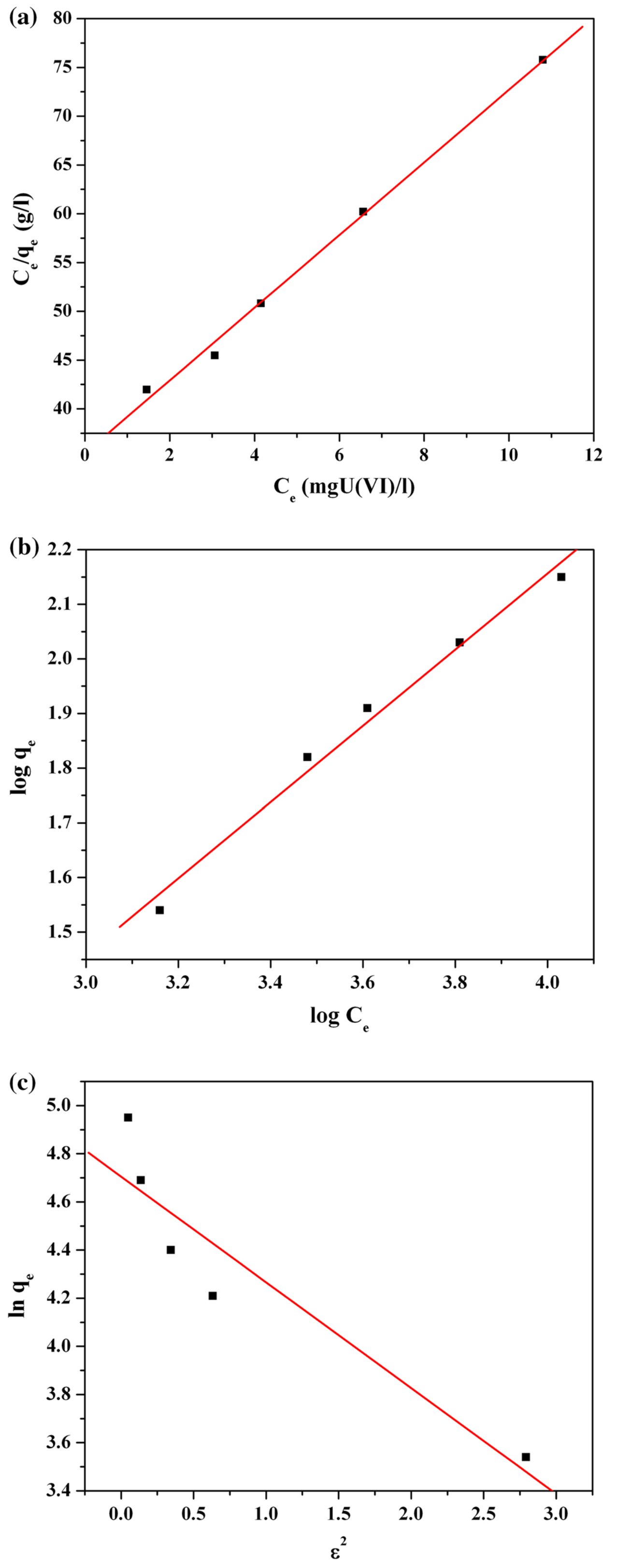

Fig. 2 Adsorption isotherms of U(VI) onto Dowex-Marathon, with 0.1 M initial concentration of UO_2Cl_2 solution, at 20 °C: **a** Langmuir isotherm, **b** Freundlich isotherm, **c** D–R isotherm

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{1}{Q_0} C_e \tag{4}$$

where C_e is uranium concentration at equilibrium (mg/l); q_e is the amount of uranium adsorbed per mass unit of Dowex-Marathon at equilibrium (mg/g); Q_0 (mg/g) and b (l/mg) are the Langmuir constants related to the adsorption capacity and rate of adsorption. The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor R_L that is given by following equation:

$$R_L = \frac{1}{1 + b\,C_0} \tag{5}$$

where C_0 is the highest initial concentration of U(VI) solution (mg/l).

The value of R_L indicates the shape of isotherms to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$) [18]. The value of R_L was found to be 0.287, which means that the shape of isotherms is favorable.

Freundlich isotherm (Fig. 2b) is based on adsorption on a heterogeneous surface and is given as:

$$q_e = K_F C_e^{1/n} \tag{6}$$

where K_F and n are Freundlich constants, which represent adsorption capacity and adsorption intensity, respectively [15]. The values of K_F and n can be determined from the intercept and slope of the linear plot log q_e versus log C_e. In our case, the slope $1/n$ was 0.697. A value for $1/n$ below one shows a normal Langmuir isotherm because it becomes more and more difficult to adsorb additional adsorbate molecules at higher adsorbate concentrations [19], while $1/n$ above one indicates a cooperative adsorption [20].

The Dubinin–Radushkevich (D–R) isotherm (Fig. 2c) assumes that the ionic species bind first with the most energetically favorable sites and that multilayer adsorption then occurs. The linear form of D–R isotherm is:

$$\ln q_e = \ln q_m - K\varepsilon^2 \tag{7}$$

where q_m is the maximum sorption capacity, K is a constant related to the mean free energy of adsorption per mole of the adsorbate, ε is the Polanyi potential ($\varepsilon = RT \ln (1 + 1/C_e)$), R (8.314 J/mol K) is the gas constant, and T is the absolute temperature [7]. The constant K gives an idea about the mean free energy (E) of adsorption per molecule of the adsorbate when it is transferred to the surface of the solid from infinity in the solution and can be calculated from the Eq. (8):

$$E = \frac{1}{\sqrt{2K}} \tag{8}$$

Table 2 Comparison of adsorption isotherms parameters of U(VI) onto Dowex-Marathon resin

Langmuir isotherm			Freundlich isotherm			D–R isotherm		
Q_0 (mg/g)	b (l/mg)	R^2	K (mg/g)	n	R^2	K (mol^2/kJ^2)	q_m (mg/g)	R^2
268.81	1.04×10^{-4}	0.9955	0.232	1.43	0.9883	0.43941	110.55	0.8715

The value of sorption energy E is 1.066 kJ/mol, which is not within the energy range of ion exchange reactions i.e. 8–16 kJ/mol, indicating the type of adsorption of uranium on Dowex-Marathon as a simple physical adsorption [21].

The parameters of adsorption isotherms of U(VI) onto Dowex-Marathon are listed in Table 2.

The highest correlation factor (R^2) was found in the case of Langmuir isotherm, which means that the U(VI) adsorption occurs with monolayer coverage onto Dowex-Marathon surface.

Kinetic data

The effect of contact time (1–60 min), initial concentration of U(VI) (0.02–0.1 M) and temperature (20–60 °C) on U(VI) adsorption onto Dowex-Marathon resin was investigated.

The procedure of kinetic experiments was the same to that of equilibrium experiments. In order to investigate the mechanism of adsorption and rate controlling steps, the kinetic data were analyzed using Lagergren pseudo-first order, Ho pseudo-second order, Weber and Morris intraparticle diffusion and Elovich equation models.

To distinguish kinetics equations based on concentrations of solution from adsorption capacities of solids, Lagergren's first order rate equation has been called pseudo-first order [22].

The rate constant of adsorption is determined from the pseudo-first order equation, given as:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t \tag{9}$$

where q_t and q_e (mg/g) are the amounts of U(VI) sorbed at time t and at the equilibrium, k_1 (1/min) is the rate constant of the pseudo-first order sorption [23]. The rate constant is obtained from *log* (q_e $-q_t$) versus t slope and q_e from the intercept (Fig. 3a).

The results of the kinetic models are shown in Fig. 3 and Table 3.

The pseudo-second order equation, based on equilibrium adsorption, is expressed as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{10}$$

where k_2 (g/mg min) is the rate constant of the pseudo-second order sorption. The linear plot of t/q_t versus t gives $1/q_e$ as the slope and $1/k_2\ q_e^2$ as the intercept (Fig. 3b).

The initial rate of adsorption h, (mg/g min) is expressed by Eq. (11):

$$h = k_2 q_e^2 \tag{11}$$

In the pseudo-second order kinetic model, the linear plot of t/q_t versus t, shows a better agreement between the experimental and the calculated q_e values than in the case o pseudo-first order kinetic model. Besides, the best fit between the kinetic models is assessed by the linear coefficient of correlation (R^2) and non-linear Chi square test (χ^2). The Chi square test measures the difference between the experimental and model data. The mathematical form of this can be expressed as:

$$\chi^2 = \sum \frac{(q_{e,exp} - q_{e,calc})^2}{q_{e,calc}} \tag{12}$$

where $q_{e,exp}$ and $q_{e,calc}$ (mg/g) are the experimental and calculated adsorption capacities from the model. If data from model are similar to experimental data, χ^2 will be small [24].

The lower χ^2 value of 3.69 for the pseudo-second order model also suggests that U(VI) adsorption onto Dowex-Marathon follows the pseudo-second order kinetics. The pseudo-first order model exhibited higher χ^2 values (55.21) suggesting poor pseudo-first order fit to the experimental data. A predominantly pseudo-second order kinetic model suggests that the chemisorption step might be rate determining and controlling the adsorption processes. The chemisorption implies exchange of electrons between the resin and U(VI) ions [15]. The pseudo-second order kinetic model is most likely to involve chemical interactions as strong as covalent bonding. The pseudo-second order kinetic analysis reveals that the values of the initial adsorption rates (h) increase with an increase in the initial concentration of U(VI), but the rate constant (k_2) decreases with an increase in initial U(VI) concentration (Table 3). The reason for this behavior can be attributed to the lower competition for the sorption surface sites at lower concentration. At higher concentration, the competition for the surface active sites is high and, consequently, lower sorption rates are obtained [25].

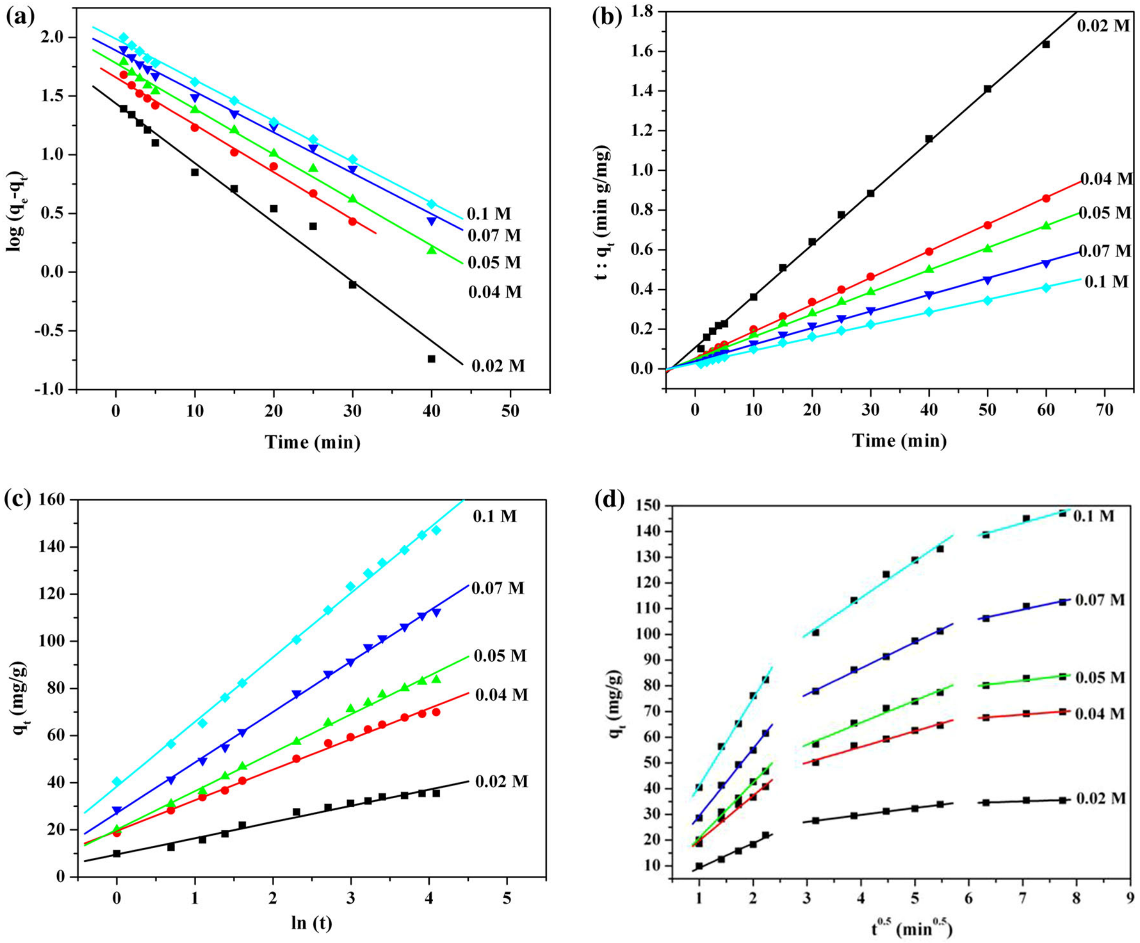

Fig. 3 Kinetic plots for the adsorption of U(VI) onto Dowex-Marathon resin. **a** The pseudo-first order model, **b** the pseudo-second order model, **c** the Elovich model, **d** the intraparticle diffusion model, for 0.02–0.1 M UO_2Cl_2, at room temperature

The Elovich equation is given in the form [10]:

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t \tag{13}$$

where α is the initial sorption rate (mg/g min) and β is related to the extent of surface coverage and activation energy for chemisorption (g/mg) (Fig. 3c).

The Elovich kinetic model is in good agreement with the pseudo-second order kinetic and describes chemisorption [26]. The values of α (Table 3) indicates that the adsorption occurs well at higher concentrations of initial solutions.

The pseudo-second order and the Elovich kinetic model could not identify the diffusion mechanism and the kinetic data were analyzed by using the intraparticle diffusion model.

Weber and Morris introduced an expression to obtain the diffusion rate coefficient, k_i, for the intraparticle diffusion kinetic model [27]:

$$q_t = k_i \cdot t^{0.5} \tag{14}$$

The rate coefficient, k_i (mg/g min$^{0.5}$) was obtained from the linear plot of q_t versus $t^{0.5}$ (Fig. 3d). In the case of a linear plot of q_t versus $t^{0.5}$ and if the line passes through the origin, intraparticle diffusion is the only rate-controlling step. If not, some other mechanisms are also involved [28].

From Fig. 3d, the multilinearity of this plot, suggests that adsorption occurred in three steps. The initial step represents surface or film diffusion, the second one represents a gradual adsorption stage where intraparticle or pore diffusion is rate limiting and the third indicates that the equilibrium is reached. The intraparticle diffusion rate constant k_i was calculated from the slope of the second linear step [25]. Similar results were reported by others [26, 27].

The pseudo-second-order rate constant of U(VI) adsorption is expressed as a function of temperature by Arrhenius type equation [28]:

$$\ln k_2 = \ln A - \frac{E_a}{RT} \tag{15}$$

Table 3 Kinetic data for U(VI) adsorption onto Dowex-Marathon resin

Parameters	Initial concentrations of UO_2Cl_2 solution				
	0.02 M	0.04 M	0.05 M	0.07 M	0.1 M
$q_{e,exp}$ (mg/g)	34.69	67.34	81.66	108.96	142.5
Pseudo-first					
$q_{e,calc}$ (mg/g)	27.23	45.47	60.06	76.98	96.83
k_1 (min^{-1})	0.1164	0.0928	0.089	0.080	0.0803
R^2	0.9776	0.9933	0.9948	0.9923	0.9974
Pseudo-second					
$q_{e,calc}$ (mg/g)	38.50	74.02	89.36	119.61	155.76
k_2 (g/mg min)	6.26×10^{-3}	3.40×10^{-3}	2.48×10^{-3}	1.78×10^{-3}	1.46×10^{-3}
h (mg/g min)	9.27	18.62	19.80	25.46	35.42
R^2	0.9990	0.9989	0.9988	0.9977	0.9979
Elovich eq.					
α (mg/g min)	27.58	55.98	58.98	76.16	111.48
β (g/mg)	0.1453	0.0769	0.0613	0.0467	0.0365
R^2	0.9776	0.9946	0.9951	0.9979	0.9970
Intraparticle diffusion					
k_i (mg/g $min^{0.5}$)	2.6854	6.1146	8.5598	10.0935	14.2182
R^2	0.9948	0.9770	0.9792	0.9959	0.9812

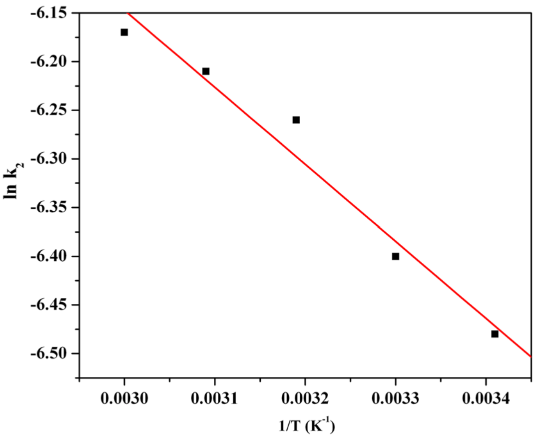

Fig. 4 Arrhenius plot for adsorption of U(VI) onto Dowex-Marathon resin

where E_a is the Arrhenius activation energy of adsorption, A, the Arrhenius factor, R, the gas constant and T is the temperature. When ln k_2 is plotted versus $1/T$ (Fig. 4), a straight line with slope E_a/R is obtained. Low activation energies (5–40 kJ/mol) are characteristics for physisorption and the predominance of van der Waals forces, while higher activation energies (40–800 kJ/mol) suggest chemisorption [29]. The magnitude of the activation energy provides information on the mechanism of the adsorption process. The activation energy of adsorption is 6.62 ± 0.16 kJ/mol, indicating that the adsorption has a low potential barrier and corresponds to a physisorption, who involves weak bonding between the adsorbate and adsorbent and this result was also in agreement with the value of the mean free energy E (1.066 kJ/mol) obtained from D–R model, which also indicates a physical adsorption. Özcan A et al. [21] studied Acid Blue 193 onto sepiolite, Ho YS et al. [29] studied the lead ion sorption on palm kernel fibre, Kilislioglu A et al. [30] studied the uranium adsorption on amberlite IR-118 H resin, and all of them reported a physical adsorption in their papers.

Thermodynamic data

In order to determine the thermodynamic parameters of U(VI) adsorption, experiments were performed at 20–60 °C. To calculate the free energy of the adsorption $\Delta(G^\circ)$, the following equation is given:

$$\Delta G^\circ = -RT \ln K_C \tag{16}$$

where K_C is the equilibrium constant

$$K_C = \frac{q_e}{C_e} \tag{17}$$

The adsorption free energy $\Delta(G^\circ)$ (Table 4) has negative values in our experimental conditions, showing the spontaneity of the process.

The Eq. (18), allows to evaluate the thermodynamic parameters of the adsorption by plotting ln K_C versus $1/T$; the enthalpy $\Delta(H^\circ)$ and the entropy $\Delta(S^\circ)$ were obtained from the slope and intercept from Fig. 5

Table 4 Thermodynamic parameters for the adsorption of U(VI) onto Dowex-Marathon resin

t (°C)	K_C	*R*	Δ*G*° (kJ/mol)	Δ*H*° (kJ/mol)	Δ*S*° (J/mol K)
20	14.27	−0.97794	−6.37	14.57	71.48
30	17.08		−7.08		
40	19.28		−7.80		
50	22.42		−8.51		
60	28.28		−9.23		

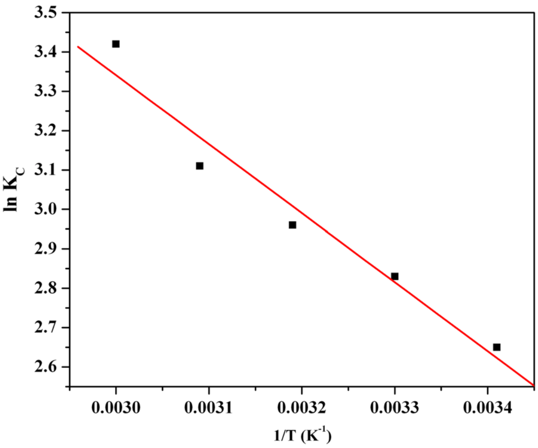

Fig. 5 Plot of ln K_C versus 1/*T* for estimation of thermodynamic parameters for the adsorption of U(VI) onto Dowex-Marathon resin

$$\ln K_C = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \tag{18}$$

The positive enthalpy shows an endothermic process. The small value of enthalpy (14.57 ± 0.36 kJ/mol) indicate that the adsorption is physical in nature involving weak forces of attraction between the resin and U(VI) ions. The positive entropy change (Δ*S*°) corresponds to an increase in the degrees of freedom of U(VI) adsorbed.

The values of thermodynamic parameters are given in Table 4.

Conclusions

The U(VI) sorption on the anion resin Dowex-Marathon is a complex process and it cannot be sufficiently described by a single kinetic model. The pseudo-second order kinetic model (chemisorption) was found to fit well with the experimental data. The intraparticle diffusion plays also an important role in the adsorption process, but it cannot be considered alone as the rate determining step of the overall process.

The equilibrium experimental data were fitted well to Langmuir adsorption isotherm.

The activation energy E_a (6.62 ± 0.16 kJ/mol), obtained from the pseudo-second order rate constant variation vs temperature, indicates a physisorption nature for U(VI) adsorption onto Dowex-Marathon resin. The small value of E_a for U(VI) adsorption onto Dowex-Marathon is in good agreement with the small E_a obtained for reaction between U(VI)$_{(R)}$ and Ti(III)$_{(S)}$. This result proves that in the rear boundary of uranium band in a separation column, at the contact between Ti(III)$_{(S)}$ and U(VI)$_{(R)}$, the rate limiting step would be U(VI)$_{(R)}$ desorption from the resin by diffusion and not the chemical reaction between Ti(III)$_{(S)}$ and U(VI)$_{(R)}$.

The positive value of enthalpy (14.57 ± 0.36 kJ/mol) indicates an endothermic U(VI) adsorption process. Although the adsorption kinetics indicates a chemisorption, the small value of the enthalpy, of the activation energy and of the energy of adsorption, denote a physisorption process. For this reasons, we suggest that de U(VI) adsorption is a physisorption process.

The Δ*G*° values were negative in the temperature range (20–60 °C) therefore the adsorption is spontaneous.

The positive value of Δ*S*° suggest an increasing of randomness at the solid/solution interface.

In the future work, we will try to measure the reaction rate between U(IV)$_{(S)}$ and Fe(III)$_{(R)}$ which, as we mentioned, takes place in the front of uranium band, in ^{235}U separation column.

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